IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Application of

Applicant(s) Serial No.

: Phelps, et al. : 10/625,915

Filed : July 23, 2003 Title

: NON-TOXIC CORROSION-PROTECTION CONVERSION

COATS BASED ON RARE EARTH ELEMENTS Docket No. : UVD 0280 IA / UD 268

Examiner

: L. Zheng Art Unit : 1742

Commissioner for Patents P.O. Box 1450 Alexandria, VA, 22313-1450

Sir:

DECLARATION OF JEFFREY A. STURGILL

Jeffrey A. Sturgill, one of the applicants in the above-identified patent application. declares as follows:

- I received a B.S degree in Geology from the University of Toledo in 1986. I was employed by the University of Dayton from November 1993 until September 2006. I have been working the area of corrosion-inhibiting pigments since 1996, and in the area of corrosion/materials degradation since 1985.
- I am familiar with this application as well as the Office Action mailed September 10, 2007, including the rejections made by the Examiner therein. I am also familiar with the references cited by the Examiner in that Office Action including U.S. Patent No. 6,200,672 to Tadokoro (treated as equivalent to WO 98/48075).
- I previously prepared solutions using Tadokoro's process and the organic compounds 2-hydroxynicotinic acid, catechol, dextrose (as a surrogate for γ cyclodextrin) and salicylic acid (slightly less soluble than 2-hydroxynicotinic acid) using

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the procedure set out in Tadokoro. See Declaration of Jeffrey A. Sturgill filed with the Amendment of June 26, 2007.

- The solubility of the dextrose-containing solution was not determined because it was too high as discussed in the Declaration of Jeffrey A. Sturgill filed with the Amendment of June 26, 2007.
- 5. The catechol/ammonium cerium IV nitrate reaction was repeated because there was not enough product (which was almost entirely pure carbon) from the earlier reaction on which to perform the solubility test. The reaction was carried out in the same way as described in Declaration of Jeffrey A. Sturgill filed with the Amendment of June 26, 2007.
- 6. The solid reaction products prepared in accordance with Tadokoro were evaluated for their solubility characteristics. The three solid reaction products evaluated included:

 salicylic acid/ammonium cerium IV nitrate;
 and 3) 2-hydroxynicotinic acid/ammonium cerium IV nitrate.

The samples were prepared for solubility determination in a manner similar to that described in ASTM D-2448: Water-Soluble Salts in Pigments by Measuring the Specific Resistance of the Leachate of the Pigment. This specification describes weighing a mass of the pigment, and then contacting the mass of powdered pigment with nine times the mass of deionized water. In the specification, the specific resistance of the 'extracting' deionized water sample placed in contact with the pigment is then determined, in order to measure how many ions were placed into the water from the pigment. This allows for a determination of the Total Salts being solubilized by the water - in effect, measuring the total solubility of the pigment in water. For this effort, that information is only part of what is needed. A measure of the cerium being extracted from the pigment/resultant solid was needed. Therefore, the extracting water sample was analyzed by inductively coupled plasma (ICP) spectroscopy in order to derive the quantity of soluble cerium in each sample.

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The ICP results for each sample are attached. Specifically, Sample 1 (reaction product of ammonium cerium IV nitrate and salicylic acid (Exhibit 1)) indicates a quantity of extracted cerium corresponding to an average of 1071 ppm (0.1 wt. %). Sample 2 (reaction product of ammonium cerium IV nitrate and catechol (Exhibit 2)) indicates a quantity of extracted cerium corresponding to an average of 6.5 ppm (0.0001 wt.%). Lastly, Sample 3 (reaction product of ammonium cerium IV nitrate and 2-hydroxynicotinic acid (Exhibit 3)) indicates a quantity of extracted cerium corresponding to an average of 81610 ppm (8.16 wt. %). As can be seen from the ICP data, smaller concentrations of other elements were detected.

Based upon a molecular weight for cerium of 140.1, these extracted concentrations correspond to cerium solubilities of:

Sample 1: 7.1 x 10-3 moles/liter

Sample 2: 4.3 x 10-5 moles/liter

Sample 3: 5.8 x 10-1 moles/liter

Tadokoro reported the solubility for the catechol and 2-hydroxynicotinic acid complexes as 0.01 mol/l or less.

- 7. The cerium content of the catechol/ammonium cerium IV nitrate reaction product is extremely low (0.0001 wt.%). The measured cerium content is probably some residual, reduced starting material. The reaction product was previously tested to be almost pure carbon. See Declaration of Jeffrey A. Sturgill filed with the Amendment of June 26, 2007.
- 8. The portion of the extract for all three solid materials that was not used for ICP analysis was then subjected to redox determination. This analysis was previously described in Paragraph 12 of the Declaration of Jeffrey A. Sturgill filed with the Amendment of June 26, 2007, but without the digestion procedure described because the extract was already in liquid form. Specifically, the titration was the procedure described on page 246 of Reagent Chemicals Specifications and Procedures 10th by the ACS

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Committee on Analytical Reagents, 2006. The 9:1 water/solid extract described above was titrated with a 0.1 N ferrous sulfate solution in the presence of a redox probe in order to determine if the cerium present was trivalent or tetravalent. These titration curves for Samples 1 through 3 indicate no change in oxidation state of the cerium in the presence of the ferrous reducing agent. See Exhibits 4-6. Therefore, there is no cerium (IV) present.

- Tadokoro does not describe having performed any procedure to determine the valence of the rare earth metal in the complex formed by the process described there.
- 10. The process described in Tadokoro is non-enabling for making a tetravalent cerium complex, and it would take undue experimentation to produce a tetravalent cerium complex using Tadokoro's process.

The declarant further states that the above statements were made with the knowledge that willful false statements and the like are punishable by fine and/or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of this application or any patent resulting therefrom.

Date: 1-9-07

Jeffrey A. Sturgil

DEC-10-2007 MC	ON 03:13 PM GFS CHEMICALS	FAX NO. 8142251175	H.
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67.0 39.0 162.9 1.7 17.8 137.7 610.2 10.9	227 171.7 171.7 73.3 20.8 32.7 58.8	0.5 1.0 56.0 21.0 28.5 75.4 75.4 75.4 75.4 75.4 75.4 75.4 75	75.0 39.0 66.5 1.3 1.3 1.8
0.001089 0.003231 0.003556 0.002084 0.000735 0.006181 0.003127 0.010430	0.000539 0.000432 0.000114 0.000114 0.001439 0.001394 0.001314 0.00236 0.00206 0.00206	0.004571 0.016864 0.00578 0.000571 0.000213 0.000213 0.000324 0.000324 0.000324 0.0001189 0.0001188 0.001188	0.001041 0.004695 0.000652 0.000032 0.000018 0.000018
udd		wada wada wada wada wada wada wada wada	undd undd undd undd undd undd
0.008277 0.002182uv 0.124590 0.004138 0.004489uv -0.000513uv 0.095353	0.015911 0.015867 0.007351 0.006820 0.006823 -0.004332av -0.00177av 0.015970 0.014858 -0.000484av -0.000484av -0.000484av	0.961956 0.996224 0.996224 0.001020uv -0.001015uv -0.001079cuv -0.001079cuv -0.001079cuv -0.001079cuv -0.001079cuv -0.001079cuv -0.00107241 0.012012 0.0110741 0.012012 0.001105 0.001105	0.001388 0.012038 0.000979 0.002479 -0.001352uv
Ag 338,289 Al 237,312 Al 308,215 Al 394,401 Al 396,152 As 188,980 As 193,696 As 224,984 B 208,956	19 249 678 19 249 678 19 253-277 19 455-408 19 1222.81 19 1223.06 19 393.366 19 393.366 10 214.439 10 228.812 10 228.812	CG 4(07,570 CG 418,659 CG 448,659 CG 228,615 CG 228,776 CG 205,560 CG 205,560 CG 205,716 CG 207,716 CG 207,716	K 766.491 K 769.897 Li 610.365 Li 670.783 Mg 279.553 Mg 280.270

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4.35845	13.4147	32000.6	21387.2	4.18897	1.05926	2.98203	2.71141	1.32313	4.67412	2.09358	0.796063	4.70826	35.5778	2.22612	28.8556	81.8946	0.999247	1.21320	3.14338	13.2490	16,5345	4.06128	2.82953	2.89582	54.5725	59.8793	168.691	1.21575	8.94595	4.54500	1.35800	685.267	272.883	31,3931	4520.28	17.3186	23.5577	13.0925	20.2975	37.8257	8.48171	68.0610	25.7583	
7.7	2.4	7	2.6	3952.8	40.8	83.8	100.8	44.7	53.7	82.6	30.2	76.6	73.7	16.8	37.3	10.5	445.4	34.1	90.3	34.1	23.4	31.9	91.1	558.0	6.6	5.3	9.4	30.5			26.4	9.0	17	101.5	190.1	49.1	29.5	36.4	35.3		3.6			
0.000693	0.000159	0.000883	0.001794	0.001088	0.000185	0.000677	0.000828	0.004561	0.003880	0.015168	0.001670	0.001836	0.006788	0.001310	0.000571	0.003645	0.003857	0.001701	0.003177	0.021959	0.014581	0.003626	0.008235	0.004832	0.003621	0.001823	0.003222	0.001762	0.002181	0.003098	0.000324	_	0.000021	0.000151	0.002502	0.000735	0.015929	0.019152	0.017152	0.000170	0.000269	9900000	0.000266	
udd	mada	Didd	udd	mdd	udd	bban	DD	mdd	mdd	Didd	mga	DD.	uida	bbm	udd	шdd	mdd	uidd	mdd	mdd	mdd	mdd	udd	mdd	bbm	bbm	mdd	ndd	udd	udd	ppm	mdd	ndd	undd	ppm	mdd	mdd	mdd	mdd	Hdd	ppm	E dd	udd	•
-0.009045uv	-0.006621uv	0.065301	0.068574	-0.000028uv	-0.000454uv	-0.000807uv	-0.000821uv	0.010203	0.007218	0.018367	-0.005539uv	0.002398	-0.009214uv	-0.007789uv	0.001531	0.034819	-0.000866uv	-0.004993uv	0.003517	0.064413	0.062306	0.011364	0.009036uv	-0.000866uv	0.036433	0.034397	0.034174	0.005784	0.045012	-0.008765uv	-0.001224uv	-0.001985uv	-0.001787uv	-0.000149uv	0.001316uv	0.001496	0.054019	0.052646	0.048642	0.007622	0.007490	0.008242	0.005855	
Mo 204.598	Mo 284.824	Na 588.995	Na 589.592	Ni 216.555	Ni 221.648	Ni 230.299	Ni 231.604	P 177.434	P 213.618	P 214.914	Pb 182,143	Pb 220.353	Pb 283.305	Pd 229.651	Pd 340.458	Pd 360.955	Pt 177.648	Pt 203.646	Pt 214.424	S 180,669	S 181.972	Sb 206.834	Sb 217.582	Sb 231.146	Si 250.690	Si 251.611	Si 288.158	Sn 189.927	Sn 235.485	Sn 283.998	Sr 216.596	Sr 407.771	Sr 421.552	11 334,941	Ti 336.122	Ti 337.280	W 207.912	W 209.475	W 220.449	Zn 202.548	Zn 206.200	Zn 213.857	Zr 339.198	

Cd 214,439

Ce 407.570 Ce 446.021

Ba 493.408 Ca 393,366 Cd 226.502 Cd 228.802 Ce 418.659 Co 230.786 Co 238.892 Cr 267.716 Cu 213.598 Cu 324.754 Cu 327,395 Fe 259.940

Bi 223.061

Ba 233.527 Ba 455.403 Bi 222,821 Ca 396.847

B 249.772

Co 228.615

Cr 205.560 Cr 206.158 Hg 184.887 HB 194.164

Fe 234.350 Fe 238.204 Hg 253.652

Weight: 0.41 Ag 338.289

abel Al 237.312 Al 308.215 Al 394.401 AI 396.152 As 188.980 As 193.696

4g 328.068

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B 208.956 B 249.678

0.218063 ppm Sc 361 383												-														0.629638 ppm Sc 361.383	-29.8492 ppm Sc 361,383		0.339003 ppm Sc 361.383	0.485030 ppm Sc 361.383					0.327768 ppm Sc 361.383		-1.07773 ppm Sc 361.383								
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0.000694	0.001281	0.004079	-0.000871uv	-0.000884uv	-0.000801uv	-0.011445uv	-0.011286uv	-0.008988uv	0.010173	0.006590	-0.001096uv	0.000124uv	-0.001060uv	-0.000371uv	0.002624uv	-0.000950uv	-0.011222uv	0.000736uv	-0.001255uv	-0.003654uv	-0.000613uv	-0.000555uv	0.000455uv	-0.000670лv	-0.005522uv	0.002582uv	-0.122382uv	-0.129521uv	0.001390uv	0.001989uv	-0.01 1998uv	-0.001436UV	-0.001000UV	-0.001/9/luv	0.00134400	0.020424	-0.004419uv	-0.001041uv	-0.002284uv	-0.002033uv	-0.000544uv	-0.001479uv	0.001327	0.002611	-0.003569uv
CCC. C12 SIM	Mg 280.270	Mg 285.213	Mn 257.610	Mn 259.372	Mn 294.921	Mo 202.032	Mo 204.598	Mo 284.824	Na 588.995	Na 589.592	Ni 216.555	Ni 221.648	Ni 230.299	Ni 231.604	P 177.434	P 213.618	P 214.914	Pb 182,143	Pb 220.353	Pb 283,305	Pd 229.651	Pd 340.458	Pd 360,955	Pt 177.648	Pt 203.646	Pt 214.424	S 180.669	S 181.972	50 200.634	28211.382	50.251.140	Si 251,090	6: 286 159	Sn 180 027	176.401 110	50 23.485	Su 283.998	Sr 216.596	Sr 407.771	Sr 421.552	11334,941	Ti 336.122	11 337.280	W 207.912	W 209.475

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Ag 328.068	0.026105	udd	0.001287	4.9	358.221	26.1050 mm	4	
Ag 338.289	0.025171		0.000754	3.0	43.3597	25.1714 mm	Se 361 383	
AI 237.312	0.028340		0.003601	12.7	26.4114	28.3397 mm	Se 361 383	
AI 308.215	0.057203) wdd	5.000675	1.2	296.182	57.2029 mm	Se 361 383	
AI 394.401	10.8704x	Ĭ	3.015898	0.1	53876.8	10870.4 ppm	Sc 361.383	
Al 396.152	0.103606) udd	0.014992	14.5	2096.74	103.606 ppm	Sc 361.383	
As 188.980	-0.009494uv) wdd	0.005764	60.7	1.49754	-9.49421 ppm	Sc 361.383	
As 193.696	-0.007416uv) udd	0.004732	63.8	0.687385	-7.41614 ppm	Sc 361.383	
As 234.984	0.210811) mdd	0.015360	7.3	35.5309	210.811 ppm	Sc 361,383	
B 208.956	0.024253) udd	.003583	14.8	7.04414	24.2528 ppm	Sc 361.383	
B 249.678	0.026916) undd	0.000854	3.2	99.3344	26.9164 ppm	Sc361.383	52
B 249.772	0.029638) udd	0.000645	22	212.957	29.6380 ppm	Sc 361.383	
Ba 233.527	-0.000854uv	•	0.000265	31.0	. 12.9036	-0.853843 ppm	Sc 361.383	
Ba 455.403	-0.001370uv) wdd	0.000041	3.0	360.875	-1.37040 ppm	Sc 361.383	
Ba 493.408	-0.000271uv	udd	1.00000.0	33,5	756.516	-0.270828 ppm	Sc 361.383	
Bi 222.821	-0.016004uv	_	0.005075	31.7	3.88297	-16.0039 ppm	Sc 361.383	
Bi 223.061	0.003142) udd	0.002084	663	4.92885	3.14230 ppm	Sc 361.383	
Ca 393.366	0.143266	•	3.000575	0.4	83101.0	143.266 ppm	Sc 361.383	
Ca 396.847	0.007374	•	0.000108	1.5	15119.3	7.37360 ppm	Sc361.383	
Cd 214.439	0.000144	_	0.000118	81.9	14.2006	0.144496 ppm	Sc 361.383	
Cd 226.502	0.001022	_	.000111	10.8	31.6891	1.02227 ppm	Sc 361,383	
Ca 407 570	0.001714	•	0.000218	12.7	31.1482	1.71449 ppm	Sc 361.383	7 0.7
0.0118.650	AUC#U.10		0.091640	3 6	001087	81045.0 ppm	Sc 361.383	
Ce 446 021	82 9391×	mdd	792074	9 0	241450	80846.2 ppm	Sc 361.383	20 TO A
Co 228 615	0.01010		100000	9 5	201200	mdd 1.88828	20,361,383	ا ا
Co 230 786	0.001210		0.00000	5/3	9.54536	1.20973 ppm	Sc 361.383	=
Co 238 807	0.004003		000000	- 1	877577	4.00253 ppm	Sc 361.383	
Cr 205 560	0.008068		900000	9.7	0.705078	-0.739554 ppm	Sc 361.383	
C-204 150	0.00000		800000	5	13.5205	8.06833 ppm	Sc 361.383	
C-263.138	0.00100	•	77,000,72	5.6	5.06906	7.59248 ppm	Sc 361.383	
Cr 267./16	0.005238		3.000170	33	66.5452	5.23759 ppm	Sc 361.383	* EXHIBIT
Cu 213.398	0.014996		000718	8.	29.4406	14.9956 ppm	Sc 361.383	Auto(n
CL 324.734	VUC/6100:0-	_	0110007	2.6	46.6102	-1.97452 ppm	Sc 361.383	er)
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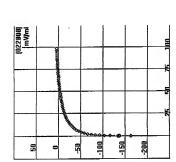
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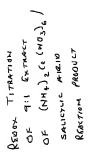
																	40																								200)						
200	50 361.383	Sc 361.383	Sc 361,383	Sc 361.383	Sc 361.383	Se 361 282	Se 361 292	Se 261 202	0.101.303	50, 301, 383	SC 361,383	Sc 361.383	Sc 361.383	Sc 361.383	56 361.383	30,301,383	Sc 361.383	50 361.383	Sc 361.383	30 301.383	Sc 361.383	Sc 361.383	Sc 361.383	Sc 361.383	Sc 361.383	Sc 361.383	Sc 361.383	Se 367.383	Se 361.383	Sc 361 383	Se 361 383	Sc 361.383	Sc 361,383	Sc 361.383	Sc 361.383	Sc 361.383	Sc 361.383	Sc 361 383	Sc 361.383	Sr 361 382	20170170	5-261.363	5-761.383	50, 100, 363	50 361.383	50 301.383	Sc 361.383	· × · · · · · · · · · · · · · · · · · ·
23/250	2.34230 ppm	-3.80120 ppm	-1.12060 ppm	-26.2798 ppm	2.13314 ppm	0.072558 rnm	6 67586 mm	2 19974 mm	A DAMON TOTAL	mqq +0++0.F	0 194040	0.104040 ppm	mdd 00106.1	7.02147 ppm	mdd /+1/2/-	and correct	74.7943	14.7043 ppm	1 19592	mdd cocoi-i-	4.18806 ppm	-u-IU3/3/ ppm	36.8598 ppm	5.87969 ppm	23.1145 ppm	36.3252 ppm	35.7528 ppm	-14.3803 ppm	-25.6118 ppm	80.7439 ppm	3056.31 ppm	0.092131 ppm	397.415 ppm	11.8510 ppm	-32.1826 ppm	-12.4820 ppm	0.837360 ppm	4.13309 ppm	85.7720 ppm	308.348 mm	293 107 ppm	364 334 ppm	und recrus	92 6497 ppm	39 9079 pm	1 10.00 a 20.00 t	-1.19299 ppm 3 58799 mm	A JAN 1940 TANDA
2 43393	2 22002	-2.32963	449.47]	12900.8	105.009	5616.92	2263.75	328.486	91 7467	74.2138	65 7406	005/-00	4 33455	6 30040	21 2080	171003	14040.5	41 0020	0200715	15.000	0.600.01	4.03382	3.60943	4.33868	2.36318	3.54721	30.1130	32.5731	-6.88811	295.645	5707.18	1.06155	55.4158	6.91495	9.89024	13.1173	1.72372	1.71475	24.9653	327.665	375 631	944 874	0.0087	14 8247	77.8570	1 45407	814180	
313	2.50	2.5	0.6	797	50.5	46.5	1.5	38	4.7	14.8	9 6	12.2	2	7.0	13.6	9	5	3 5	27.6	2 5	269.5	2.00	4.0	17.2	6.19	Ξ	10.5	40.2	23.0	4.7	0.2	7629.9	9.0	25.6	23.2	165.8	244.8	155.5	4.8	1.2	8.0	0.2	9	00	? =	13.0	1.7	:
0.000734	0.00000	0160000	2680000	0.007021	0.001077	0.000034	0.000099	0.000085	0.000172	0.000042	0.000114	0.0000243	0.00000	0.000735	0.000418	0.001915	0.000240	0.00000	0.000427	0.000419	0.000110	0.00000	0.001000	0.001011	0.014303	0.004048	0.003744	0.005781	0.005900	0.003781	0.007109	0.007029	0.002561	0.003033	0.007466	0.020699	0.002049	0.006428	0.004125	0.003671	0.002292	0.000672	0.003912	0.007522	0.004385	0.000155	0.000061	
mud		H	mdd	mdd	udd	udd	undd	II dd	mod	шаа				200								The state of	mdd	mdd	udd	udd	udd	udd	mdd	mdd	bbm	mdd	wdd	ppm	undid	ardid	mdd	mdd	mdd	mdd	udd	mdd	шида	maa	m did		E 60	
0.002543	-0.003801nv	0.00112 tar	-0.001121UV	-0.026280uv	0.002133	0.000073	0.006676	0.002200	0.004044	-0.000286uv	0.000184	0.001981	VIII 600000-	-0.007914nv	-0.003071uv	0.032237	0.044784	0.021337	-0.001186uv	0.004188	-0.0001061114	0 036860	0.000000	0.002880	0.023115	0.036325	0.035753	-0.014380uv	-0.025612uv	0.080744	3.05631x	0.000092uv	0.397415	0.011851	-0.032183uv	-0.012482uv	0.000837µv	0.004133uv	0.085772	0.308348	0.293107	0.364334	0.056724	0.083649	0.038893	-0.001193uv	0.003588	
104 174 104	Hg 253,652	K 766 491	7 760 B7	T: C10.097	7. 610,365	1.1 6 /0.783	Mg 279.553	Mg 280.270	Mg 285.213	Mn 257,610	Mn 259.372	Mn 294.921	Mo 202.032	Mo 204.598	Mo 284.824	Na 588,995	Na 589.592	Ni 216.555	Ni 221.648	Ni 230,299	Ni 231.604	P 177 434	P 213 618	P 214 014	Dh 102 147	Dt 220 143	F0 220.353	Pb 283,305	Pd 229.651	Pa 340.458	Pd 360,955	Pt 177.648	Pt 203.646	F1214.424	9 181,009	2161.915 Ch 200 Pa	Sh 217 692	20.717.06	Si 250 Coo	31 230.690	51.251.611	Si 288.158	Sn 189.927	Sn 235.485	Sn 283.998	Sr 216.596	Sr 407.771	

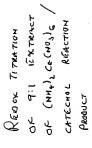
		e	%RS	SD(Int)	Int (c/s)	Rafio	Label
Sc 361.3	43.2557 ppm	1031.43	2.6	0.001119	mdd	0.043256	Zr 349.619
Sc 361.3	23.0793 ppm	543.630	1.7	0.000393	udd	0.023079	Zr 343.823
Se 361.3	6.59574 ppm	34.3309	3.2	0.000211	mdd	0.006596	Zr 339.198
Sc 361.3	1.97315 ppm	24.7303	6.7	0.000132	mdd	0.001973	Zn 213.857
Sc 361.3	7.31213 ppm	8.32183	3.0	0.000216	mdd	0.007312	Za 206.200
Sc 361.3	5.46624 ppm	29.0883	3.9	0.000212	mdd	0.005466	Zn 202,548
	The state of the s	2000000	1	*******	-	1	******

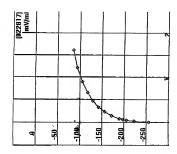
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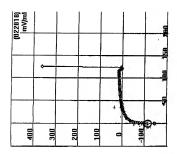
EXHIBIT











REDOX TITRATION

OF 9:1 Extenct

OF (NH4)2 Ce (NO3)6

2-HYDEOXY MATHOR ACID

REACTION

REACTION